AD-A177 009 SUPERLATTICE EFFECTS IN GRAPHITE INTERCALATION COMPOUNDS(U) NORTHEASTERN UNIV BOSTON MASS DEPT OF PHYSICS R S MARKIEWICZ 15 APR 86 AFOSR-TR-87-0104 1/1

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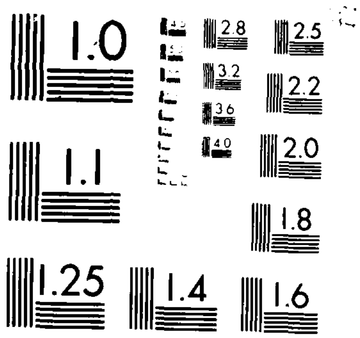


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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

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Condon domains
quantum Hall effect
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quasiperiodicity

long-chain ...

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Research is continuing into the Landau level condensation of a dense two-dimensional hole gas in a strong magnetic field, recently discovered in $C_{16}Br_2$. The series of phase transitions, periodic in (inverse) magnetic field, produce a domain phase which is an ideal candidate for the study of nonlinear soliton (domain wall) dynamics. Study of a.c. coupling to a normal mode oscillation (bending mode) of the domain wall indicates chaotic behavior following a quasiperiodic route. A simple model is capable of explaining the

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20. Abstract (cont.)

observed lineshape of the susceptibility oscillations and unambiguously determining the Landau level bandwidth -- thereby pinpointing the field at which the strong two-dimensional limit occurs (no Landau level overlap). As the sample thickness is decreased, it is predicted that the system will transform into a charged-domain phase closely analogous to the quantum Hall effect.

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1. Summary of Research Goals and Plans

i) To thoroughly examine the recently discovered Condon phase in $Br_2$ (and possibly other) intercalation compounds, both to understand the soliton (domain wall) dynamics and to explore the predicted connections between this phase and the quantum Hall effect.

ii) To use magnetooscillations and x-ray diffraction as probes to study superlattice formation in graphite intercalation compounds, particularly in those situations in which magnetic breakdown or field-induced phase transitions suggest that the superlattice may be generated by Fermi surface instabilities.

iii) To search for pressure-induced phase transitions in intercalation compounds, especially via resistivity probes.

2. Status of the Research Effort

The research on Landau level condensation (Condon domains in a two-dimensional hole gas) continues to be extremely interesting. This phenomenon is a phase transition which occurs in a two-dimensional electron (or hole) gas in a strong magnetic field, whenever the gas is near a filled Landau level. The sample breaks up into domains such that in each domain all Landau levels are either exactly full or empty. In a thick sample, such as the $C_{16}Br_2$ intercalation compounds we are studying, the magnetic field forms domains while the carrier density remains uniform. In the domain phase, the dynamics is due to soliton (domain wall) motion, and hence is highly nonlinear.

The study of nonlinear dynamics, and in particular routes to chaos, is being quite actively pursued at present. A recent trend is to move away from model systems (nonlinear capacitors) and look for nonlinear dynamical effects in more complicated systems, such as Josephson junctions or charge-density

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waves. At the March APS Meeting, four sessions were devoted to charge-density waves, and a large fract of the talks were concerned with the nonlinear dynamics of the depinned charge-density wave state. Nearly every experimental observation could be matched by an effect in the Landau level condensate, and the condensate offers a number of distinct advantages:

1. The frequencies are much lower (kHz vs MHz or GHz), so much more detailed studies can be performed -- such as the classical experiments on chaotic dynamics -- return maps, phase portraits, etc.[1]

2. The d.c. magnetic field offers a convenient parameter for varying the properties of the condensate. Small changes of field within a single Landau level can tune the resonant (bending mode) frequency of the domain wall. Since there are transitions associated with each Landau level, however, the effect of much larger changes of the parameters can also be probed. For instance, only one hole band is involved at low fields, whereas a second becomes important at higher fields. The influence of two k-space periodicities on nonlinear dynamics is an area which has so far been unexplored. (There are predictions[2,3] that this can produce a new type of condensate, a magnetization density wave.)

3. The Landau level condensation is taking place in a system that is inherently simple and thus has been the subject of considerable study, the two-dimensional electron gas.[4] This suggests that a very detailed <u>microscopic</u> picture of the

condensate should be possible, and holds out the possibility of a similar microscopic picture of the nonlinear dynamics.

A disadvantage of the condensates has been the environmental sensitivity of the samples, graphite intercalated to stage 2 with $Br_2$. Our attempts to break the samples out of sealed glass ampoules and attach electrical leads have so far proven unsuccessful. Hence we have not been able to carry out the combined a.c.-d.c. experiments which are being studied in charge-density wave systems -- e.g., the observation of Shapiro steps.[5] R. Hall (Berkeley) suggested to me at the March meeting that instead of d.c. I try the experiments with a very low a.c. frequency (~100 Hz). This simple experiment should work, and we will try it out as soon as the Magnet Lab reopens.

In addition to the inherent interest in the Landau level condensation as a phase transition in an interacting electron gas, with the associated nonlinear dynamics, there is the possibility that this phase can shed light on the quantum Hall effect (QHE). Vagner, et al.[6] predicted that this phase would share some properties of the QHE, and I suggested[7] that this magnetic domain phase may actually be a dual to the QHE. I have recently clarified the analogy, and in so doing have come up with a viable picture for the integral QHE, which clearly brings out its connection to the fractional QHE (discussed below).

Given all of these unique features, we have been intensively pursuing our study of this phase--trying to sort out a variety of problems and to develop new probes into the nature of this system:

a. Lineshape analysis. No quantitative study of the condensed phase could be undertaken until we had developed a theory capable of explaining the shape of the observed a.c. susceptibility oscillations. Six months ago the lineshapes were a total mystery -- completely diferent from our expectations. The mystery is now solved -- the presence of two hole bands allows a continual exchange of holes between the two bands as the magnetic field sweeps between Landau levels. This greatly modifies the susceptibility lineshapes. Fig. 1 shows that the lineshapes calculated from a simple two-dimensional picture[8] are in very good agreement with our observations. We see a large sample-to-sample variation of lineshapes, which can be explained as due to broadening in the heavy hole band (larger $\Gamma_2$). The lower two frames are typical of our sample-to-sample variation. The upper frame is anomalous -- it is taken from a sample which was air-exposed for lead attachment. The signal is much weaker, there are no signs of nonlinear effects associated with domain formation, yet the susceptibility oscillations are much sharper, reminiscent of the expected Fang-Stiles lineshape.[9] The reason for this is simple. The sealed samples are nearly two-dimensional, but the residual interlayer interaction gives a small bandwidth (about 5 meV) to each Landau level. This is responsible for most of the broadening in the middle frame of Fig. 1. The unsealed sample has degraded to the point where interlayer interactions are negligible, so the Landau level bandwidth is actually reduced to a residual value due to disorder broadening.

More direct evidence for the c-axis dispersion is given in Fig. 2, data similar to Fig. 1 but at lower fields. The structure within each Landau level is due to level overlap. Fig. 2a is the data, 2b the theoretical calculation, while Fig. 2c is an explanation. The susceptibility gives approximately a picture of the density of states. Because of the c-axis dispersion, the density of states is that appropriate for a one-dimensional band, with divergences on both sides. At sufficiently high fields, the cyclotron energy (Landau level separation) is greater than the width of an individual level, and the susceptibility describes isolated Landau levels. At lower fields, Landau levels start to overlap and the susceptibility, being the sum of several levels, take on quite complicated patterns. The matchup between Figs. 2a and 2b suggests that the bandwidth is about 5 meV, in excellent agreement with my earlier prediction.[10] [At the March Meeting, Zaleski and Datars[11] reported similar good agreement for the bandwidth of a different intercalation compound -- $SbC\ell_5$-graphite].

While the overall agreement between theory and experiment expressed in Figs. 1 and 2 is quite good, these are a number of differences in detail. This is to be expected, since the theory is a one-electron model which ignores domain formation. Inclusion of these condensation effects is in principle straightforward -- in the absence of dynamical effects, the susceptibility $\chi$ is cut off at $4\pi\chi = 1$, and the remaining oscillations slightly broadened. At this point, however, an unknown parameter enters the theory. While the lineshape is fixed by a noninteracting electron theory, the amplitude of the oscillations is sensitive to electron interaction.[12] The present calculations give the opposite sign from my observations. These calculations, however, ignore many effects (e.g., correlated ring exchange[13]) which are now known to be important contributions to the electron gas binding energy. Since these same contributions are essential for understanding the QHE, I believe that it would be important to have a proper calculation of these effects, and I have discussed this possibility with a number of theorists. Until such calculations exist, I can only offer approximate calculations of the susceptibility lineshapes using empirical enhancement factors.

b. Nonlinear dynamics. I had earlier noted that my observations were very similar to the quasiperiodic route to chaos studied by Libchaber's group[14] in Rayleigh-Benard convective columns of Hg. From the March meeting, I could see that this same route is appropriate for charge density waves. Very detailed studies of the same process in still different solid state systems were presented at this meeting.[15,16] The basic experimental observations, common to all studies, are as follows: A dynamical system having a well-defined characteristic frequency $f_1$ is perturbed by an external probe of frequency $f_2$. For low amplitudes of the probe signal, the two frequencies coexist independently of each other. At higher amplitudes, nonlinear effects first produce mixing frequencies (sums and differences of $f_1, f_2$), then shift $f_1$ to a nearby value commensurate with $f_2$: $f_1/f_2 = n/m$, where n and m are integers (entrainment). This entrainment follows a well-defined sequence, and as the drive amplitude is increased, the range of values $f_2$ over which $f_1$ is entrained to a particular value increases. At large enough amplitude, these entrained regions start to overlap. At this point, $f_1$ does not 'know' which value to lock into, and the system undergoes a chaotic motion. This is illustrated for Landau level condensation in Figs. 3 and 4. In this case, the natural frequency $f_1$ is due to a bending mode in a domain wall. Since the restoring force is increased by the presence of other, nearby domain walls, the frequency $f_1$ can be tuned by varying the applied magnetic field, and this is the procedure followed in Fig. 3. This figure shows the Fourier transforms of the a.c. pickup of a coil surrounding the sample. Fig.

(3a) shows a sequence of sixty traces at slightly different values of applied field. For clarity, only a limited range of frequencies around the third harmonic of $f_2$ is displayed. As the field changes, the driven oscillator is entrained successively over regions of period 3,4,5, and 6, each separated by regions of broadband noise. Fig. (3b) shows a different change in periodicity, but not accompanied by any noise. The frequency $f_1$ shifts smoothly between two commensurate values, passing through an unlocked region in which the ratio $f_1/f_2$ takes on incommensurate values. Fig. 4 shows a Fourier transform of the broadband noise regime over a wider frequency range (actually an average over 500 Fourier transforms, to smooth out the random fluctuations). The spectrum is revealed to be a very broadened version of the resonance, with a peak at the value $f_1$, a broad tail extending to zero frequency, and an exponential falloff at high frequencies. This is similar to the broadband noise lineshape observed in charge density waves[17] and in the helical instability in a semiconductor plasma[15] (insert in Fig. 4).

There are potentially four domains of chaotic response: (1) near the low-field threshold; (2) away from threshold in the light-hole band; (3) in the heavy-hole band; (4) in the mixed phase (magnetization density waves[2,3]). The most sensitive means of ditinguishing these four domains may be by observation of the strange attractor.[1] We are planning to build the required circuitry for these studies. Basically, one looks at the "return map": at any particular phase in the driven $f_2$ oscillation (e.g., the peak value) sample the amplitude of the pickup coil response $V_n$ on each (nth) cycle. A plot of $V_{n+1}$ vs. $V_n$ (the return map) will show how much correlation there is in the system. If the system is chaotic, the return map is topologically a two-dimensional section of a portrait of the strange attractor. [Since the attractor is often multidimensional, it may be an advantage that our $V_n$'s are complex.] If these experiments show distinguishable strange attractors in the four cases, it may give a clue as to the nonlinear dynamics, as well as shed light on the possible density wave. It will then be important to extend these studies to thinner samples, to look for evidence of the predicted[7] domain to vortex transition.

c. Other experiments. Work is progressing on a variety of other experiments:

- A sample stage is being built for heat capacity measurements, to search for the predicted giant magnetothermal oscillations, the characteristic oscillatory heat capacity of a two-dimensional electron gas,[18] and direct evidence for the domain phase transition.
- Bi fine wire probes have been received from IBM. These will be used to monitor domain wall motion near the sample surface, as a direct probe of the existence and dynamics of domains.
- Optical experiments are being undertaken to directly image domains via Faraday rotation (viewing the sample through crossed polaroids will detect the presence and spatial distribution of magnetic domains). We have found a broad luminescence band in the visible, due to the $Br_2$ molecules. This band shows a strong residual polarization with a sizable Faraday effect, and hence is a good candidate for these studies.
- Magnetoresistance and Hall effect studies show broad oscillations which correspond to the upper frame of Fig. 1 (they are measured simultaneously). Unfortunately, the sample surface shows even stronger

degradation than the bulk, and no effects due to domains are seen. Continuing efforts will be made to measure d.c. transport properties, including the quasi-d.c. studies mentioned above.

d. The Quantum Hall Effect. In any Condon domain, the magnetic field is fixed at the value corresponding to an exactly filled Landau level. This implies that the first excited state is separated from the filled band by a large energy, and hence that there is no resistive scattering. Hence the Condon domains in two dimensions should show a form of quantum Hall effect, with zero longitudinal resistivity.[6] This suggests that there are three independent quantum Hall effects (since the connection between the integer and the fractional QHE is obscure). I believe there is a close connection between these three effects, and I think I have found it.[7] The Condon domains are a predominantly magnetic effect, but if the sample thickness is decreased the domain phase transforms to a vortex phase which is dominated by electrical effects. This vortex phase is a dual to the ordinary domain phase: in the domain phase the carrier density is uniform, the magnetic field, bunched into domains; in the vortex phase the magnetic fields are uniform, while the carrier density forms domains. In both cases, within the domains, the Landau levels are exactly full or empty.

The vortex phase is, I believe, essentially equivalent to the integer QHE. The vortex is a domain of lower charge density, but shrunk down to the size of a single hole. The binding energy of the vortex state then reduces to the localization energy of a single hole. Hence the vortex state is stabilized by disorder, since in two dimensions all states are localized. The transport properties then are those of a uniform electron gas exactly at a filled Landau level, but containing a vortex array. The longitudinal resistance is zero, as discussed above, and the Hall effect is unchanged from its value at the center of the Hall step, since the vortices act as finite inclusions, which have no influence on the Hall effect.[21] This description fails only halfway between two Hall steps, when the vortices percolate through the sample, causing a finite magnetoresistance and a Hall resistivity which smoothly extrapolates from one Hall step to the next. Recently, some other groups have proposed similar models for the QHE.[19]

The above picture clearly shows the connection between the integer and fractional QHE. The only difference is that in one case the excitations are inter-Landau level holes, in the other they are Laughlin's quasiholes.[20] The predicted connection between the vortex phase and the QHE is the reason we are working so hard to develop probes which will allow us to directly study the domain to vortex transition, and explore the unusual properties of the vortex state.

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## Figure Captions

Fig. 1. Comparison of experimental (left) and theoretical (right) susceptibility lineshapes for several samples of $Br_2$-intercalated graphites. Top=air-exposed sample; middle and lower panels=typical sealed samples.

Fig. 2. Susceptibility lineshapes in a lower field range, showing effects of Landau level overlap: (a) Experiment; (b) Theory; (c) Explanation, showing c-axis dispersion and subsequent density of states at two field values.

Fig. 3. Fourier transform of a.c. pickup, showing effects of quasi-periodicity. Each figure shows 60 sequential transforms,each at a slightly higher value of d.c. magnetic field.

a. Region of subharmonic lockin: regions of period 3,4,5, and 6 separated by broadband noise.

b. Crossover of the locked regimes via an unlocked (incommensurate) state. Note that the mixing frequencies shift smoothly with field between two locked states.

Fig. 4. Fourier transform of region of broadband noise, showing high frequency exponential tail. Insert: Similar broadband noise spectrum, but from the helical plasma instability in Ge [From G. Held, Thesis, U.C. Berkeley 1985]

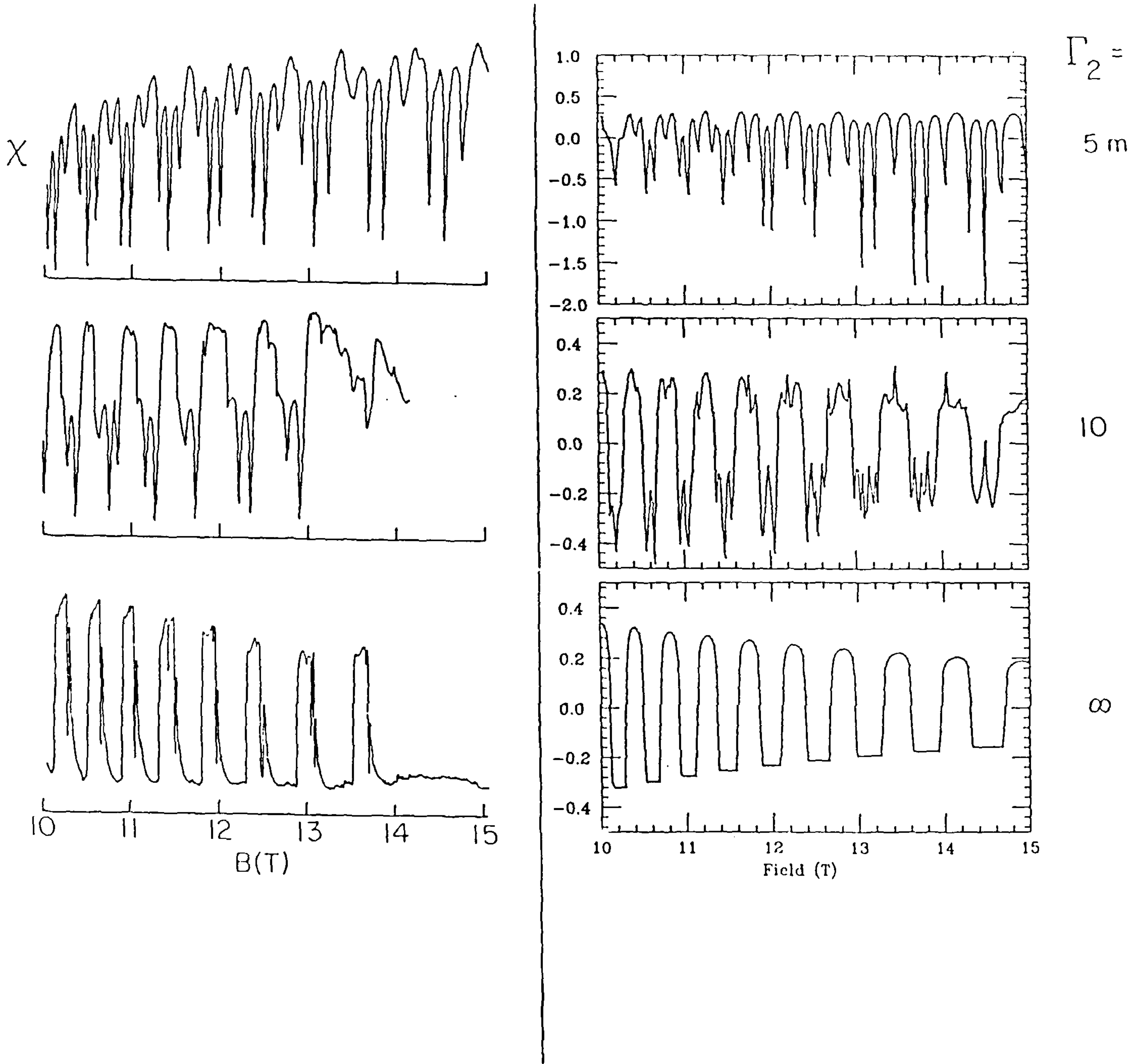
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Fig. 1

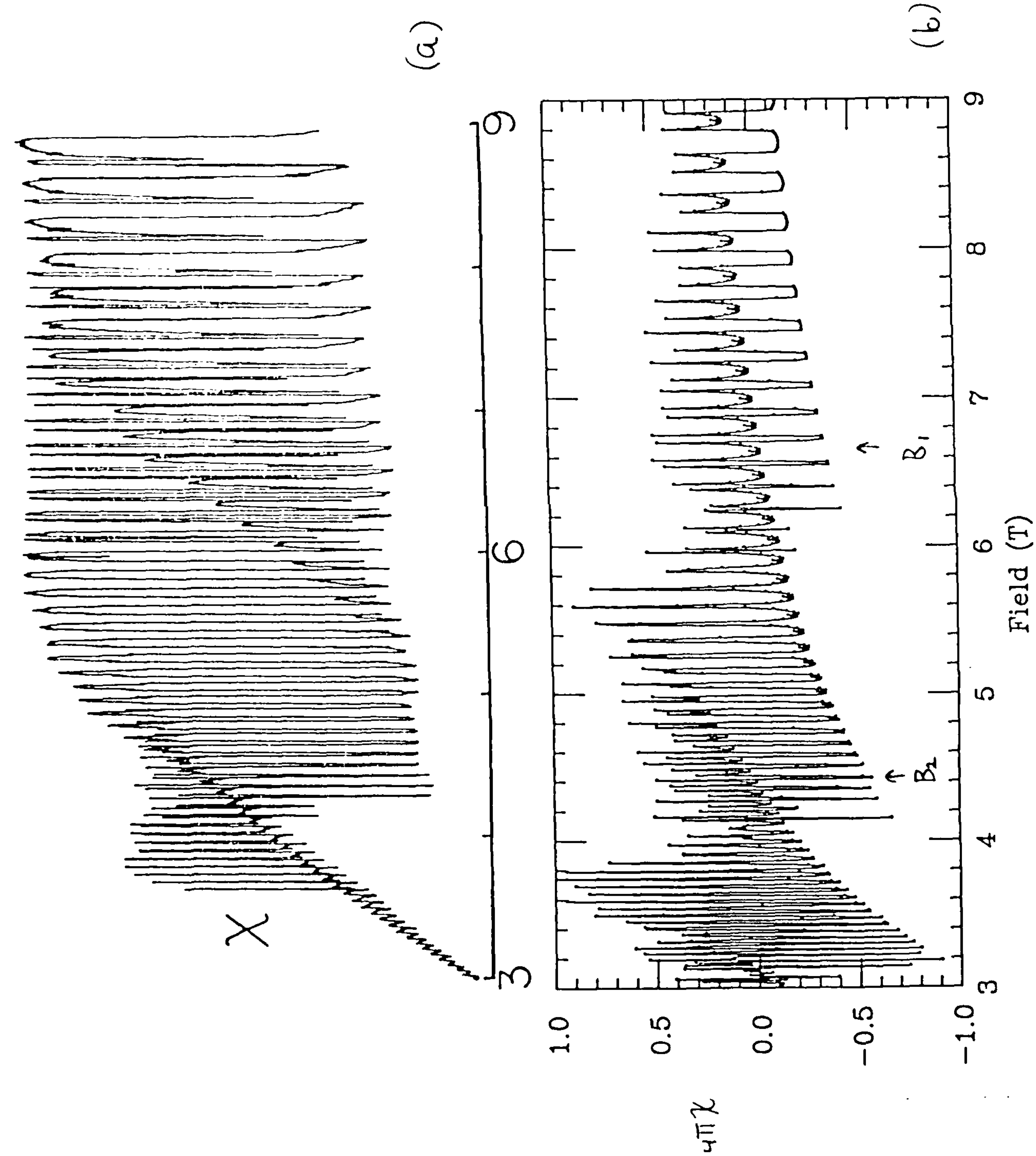



Fig. 2

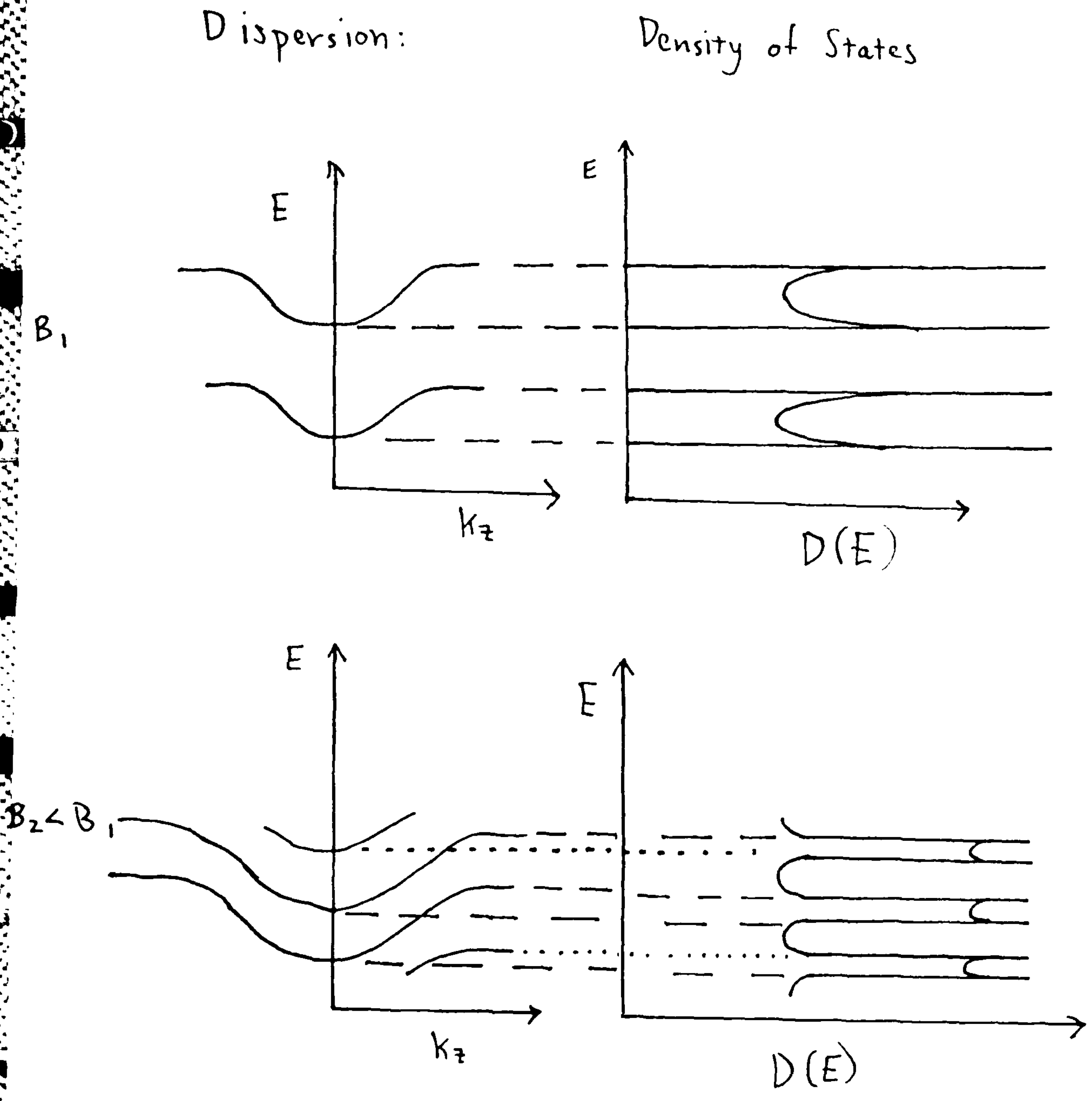
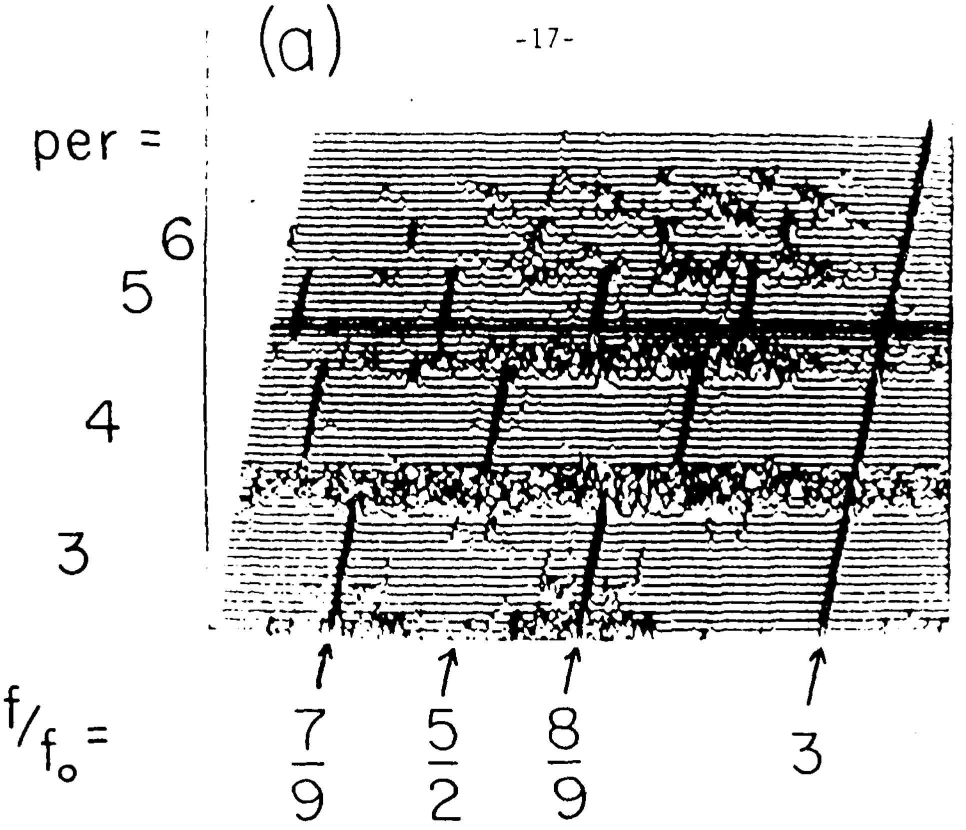



Fig. 2c



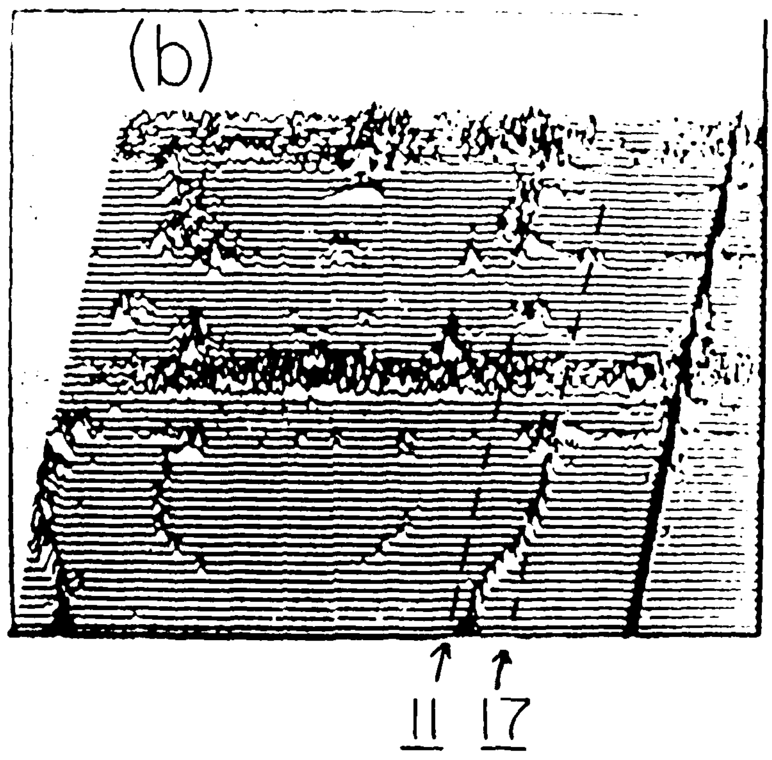
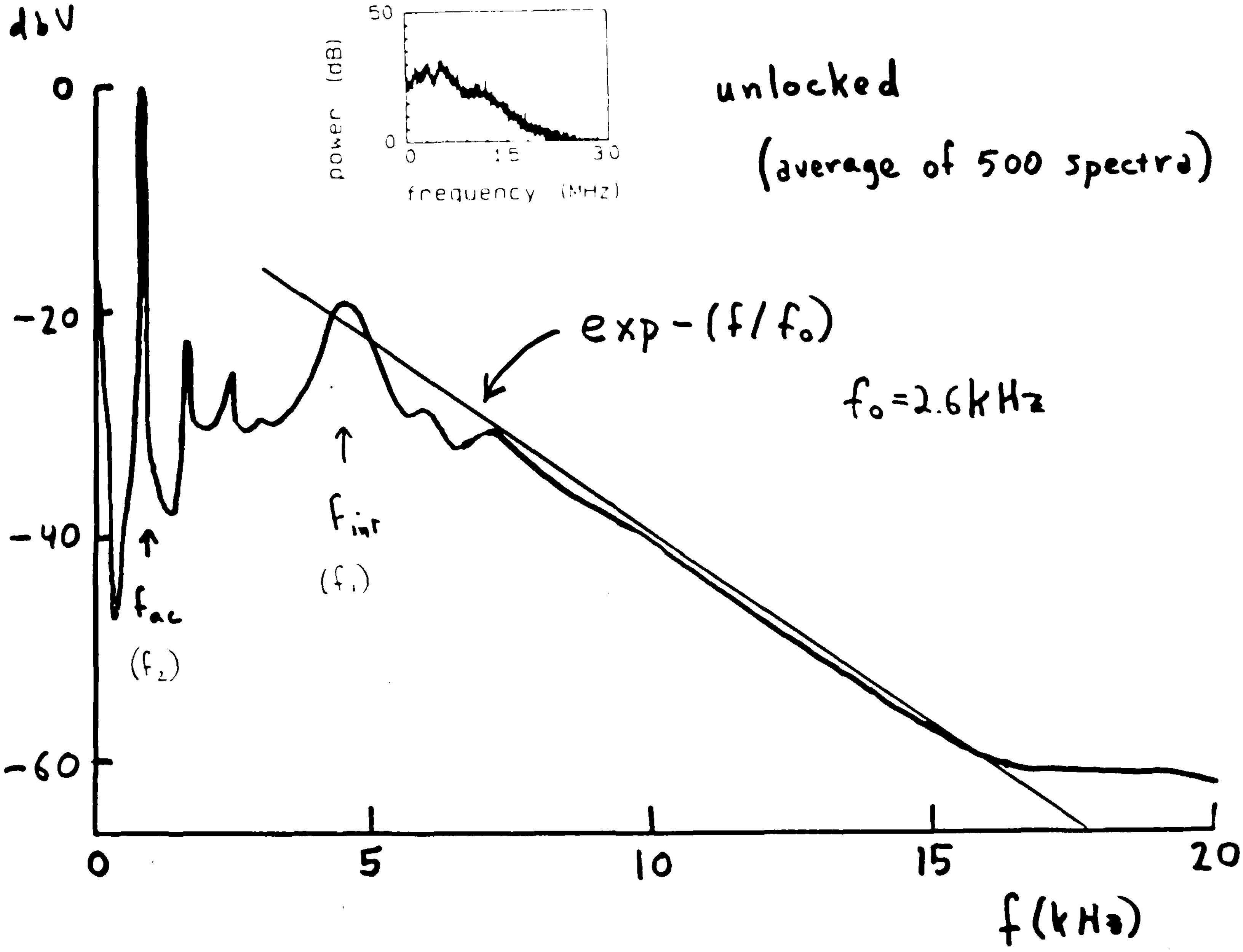




Fig. 3



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f_int
(f₁)
f_ac
(f₂)
50
0
15
30
power (dB)
frequency (MHz)


Fig 4

3. <u>Research Equipment Acquired</u>

HP 3561A Dynamic Signal Analyzer

PAR Model 113 Preamp

4. <u>List of Publications</u>


1. "Field-induced Phase Transition in $AsF_5$-graphite," R.S. Markiewicz, C. Zahopoulos, D. Chipman, J. Milliken, and J.E. Fischer in P.C. Eklund, M.S. Dresselhaus, and G. Dresselhaus, Eds. Graphite Intercalation Compounds: Extended Abstracts (Pittsburgh, Mat. Res. Soc., 1984), p. 42.
2. "Magnetic Interference and Breakdown in Intercalated Graphite," C. Zahopoulos and R.S. Markiewicz, ibid., p. 48.
3. "Magnetooscillations in Intercalated Graphite Single Crystals," M. Meskoob, C. Zahopoulos, and R.S. Markiewicz, ibid., p. 57.
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8. R.S. Markiewicz, "Condon Domains in a Two-dimensional Electron Gas, I. Domain to Vortex (Modulated) Transition," submitted to Phys. Rev. B.

9. R.S. Markiewicz, "Condon Domains in a Two-dimensional Electron Gas, II. Charging Effects," submitted to Phys. Rev. B.

10. R.S. Markiewicz, "Condon Domains in a Two-dimensional Electron Gas, III. Dynamic Effects," submitted to Phys. Rev. B.

11. R.S. Markiewicz and M. Meskoob, "Chaos in the Dense Two-dimensional Electron Gas in a Strong Magnetic Field," Bull. A.P.S. 31, 587 (1986).

5. Professional Personnel:

R.S. Markiewicz, Principal Investigator

C. Zahopoulos, Graduate Student†

M. Meskoob, Graduate Student

K. Chen, Graduate Student

B. Maheswaran, Graduate Student

L. Fotiadis, Graduate Student

X. Wu, Graduate Student (Reading Course)

M. El Rayess, Post Doc

†Received Ph.D. 3/85. Thesis "Fermiology of Acceptor Graphite Intercalation Compounds Using de Haas-van Alphen and Shubnikov-de Haas Measurements."

6. Interactions

a. Papers presented at scientific meetings:

(i) Refs. 1-3, presented at Materials Research Society Meeting, Boston, November 1984.

(ii) Ref. 5 presented at APS March Meeting, Baltimore, MD, March 1985.

(iii) Ref. 6 presented at Int. Conf. on Graphite Intercalation Compounds, Tsukuba, Japan, May 27-30, 1985 (invited).

(iv) Ref. 11 presented at APS March Meeting, Las Vegas, Nevada, March 30-April 3, 1986.

b. Seminars given or arranged:

(i) "Superlattices and Phase Transitions in Graphite Intercalation Compounds," IBM, Yorktown Heights, October 26, 1984.

(ii) "Giant Magnetic Interaction in a Two-dimensional System: Possible Connection to the Quantum Hall Effect," Northeastern Physics Dept. Colloquium, November 1984.

(iii) "Phase Transitions in Graphite Intercalation Compounds," Chemistry Dept., Northeastern, February 25, 1985.

(iv) "Giant Magnetic Interaction and Domain Dynamics in Two Dimensions," Boston University Physics Dept., May 1, 1985.

(v) Visit to J. Koplik and H. Levine, Slumberger, July 1985.

(vi) "Condon Domains and Chaos in Graphite Intercalation Compounds," Dresselhaus group meeting, MIT, September 19, 1985.

(vii) "Landau Level Condensation and Domain Wall Dynamics in Two Dimensions," Harvard Theory Lunch, January 30, 1986.

?. Collaborations

(i) Dr. David Chipman, A.M.M.R.C., Watertown Arsenal: Transmission x-ray studies.

(ii) Prof. J. Fischer, U. Penn., Philadelphia: magnetooscillations in mercurographites.

(iii) Dr. J. Milliken, NRL: magnetooscillations and x-ray studies of $AsF_5$-graphite with excess F.

(iv) Prof. R. Clarke, U. Mich., Ann Arbor: magnetooscillations and x-ray studies of single crystals of $HNO_3$-graphites.

(v) M.J. Brady, R. Webb and Dr. E. Pakulis, IBM, Yorktown Heights: formation of Bi microprobes to observe domains in $Br_2$-graphite.

(vi) Prof. J. Brooks, B.U., Boston, NMR and magnetization of $Br_2$ in $Br_2$-graphite.

(vii) Prof. L. Falicov, U.C., Berkeley: calculation of magnetic breakdown in 2-d.

(viii) Prof. G. Zimmerman and A. Ibrahim, B.U.: magnetic intercalation compounds ($FeCl_3$).

(ix) H.A. Resing and M. Rubenstein, NRL: NMR in $Br_2$-graphite.

(x) P. Sagalyn, AMMRC: high-resolution C,F NMR in $SbF_5$- and $SbCl_5$-graphites.

(xi) C. Perry and L. Reinisch, NU: optical studies of $Br_2$-graphite.

?. Patents

N.U. lawyers are doing patent search regarding possible patent on d.c. transformer based on Condon domains.

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